

FEI tecnai f20- video script

In a transmission electron microscope an electron beam from the electron gun, passes through an electromagnetic condenser lens, through the sample, through an objective lens and then down to a phosphor screen or CCD camera. The condenser lens controls the size and intensity of the beam hitting the sample. The objective lens controls the magnification.

TEM images show internal structures of specimens with magnification up to one million times revealing structures in atomic resolution.

The first step is to prepare the sample and mount it in the holder.

Remove the cover tube from the single tilt holder. Insert the tool into the hole and gently lift the clamp.

Place tissue paper beneath in case the sample falls off. This sample has gold nanoparticles on a copper grid. Place the side with the particles facing down inside the specimen carrier. Gently lower the clamp. Replace the cover tube.

Carry the sample with a hand over the end so that arm can't slide out.

Place the copper cold fingers slowly into a Dewar of liquid nitrogen. Top- off the Dewar with additional liquid nitrogen. This will keep the sample cold.

The TEM uses four pieces of software which should be opened in order.

Open tecnai user interface software PEUOil? and you will hear a "click click" which confirms that the microscope is communicating with the computer.

On the second monitor we see the DigitalMicrograph software.

The third software is TIA- "TEM imaging and analysis" which is used for recording Scanning TEM and EDS.

The last software is the RTEM Control.

Select "setup", "vacuum" and "turbo on" and hear the vacuum pump sound. After 3 minutes the "Turbo On" boxes changes to yellow.

In the user interface software the box on the lower left tells the magnification and the microscope mode, currently TEM bright field and 175,000 magnification.

To the right the beam energy and spot size are shown. While setting- up the spot size will be 5 and during operation it will be 3. The exposure time tells you the brightness and illumination of the beam.

The lower left box with L1, L2, L3s reflect the left control panel and the R1, R2 and R3 shows the state of the right control panel. The multifunction controls are used for several functions in different modes.

In the right bottom box select “vacuum overview” to show the status of the vacuum system and valves.

Now the “Turbo on” button is yellow indicating the turbo pump has reached its full function and the TEM is ready to receive the sample.

Take the holder out of the support. Place the little pin on the holder in the 5 o’clock position with a twist adjustment.

The light goes on, the valves are opened and the pump evacuates the sample chamber. You must wait for several minutes until the light goes off or you could crash the vacuum.

You must also tell the computer that it is a single tilt holder so that the holder can be controlled with a joystick.

When the light goes out and the “airlock” status changes to “column valves” the sample chamber has been evacuated.

Now turn the holder counterclockwise and hold it as the vacuum sucks the holder slowly in.

Now turn off the “turbo” pump.

After the holder has been inserted the vacuum in column should read 6 and you can start to adjust the beam.

Click the “column valves closed” and the button changes from yellow to gray and the status reads “ready”.

Take the cover off the phosphor screen.

If you don’t see a beam, decrease the magnification with the magnification dial on the right control panel.

At low magnification you see a bright spot. As you increase the magnification you can see the copper grid.

Use the joystick to move the sample.

When you change the magnification you also have to adjust the intensity to illuminate the whole field of view.

Counter clockwise condenses the beam and clockwise spreads the beam.

Next we will do alignment

Use the trackball to move the beam to the center, spread the beam, increase magnification and then condense the beam.

The rings are caused by diffraction.

Click “eucentric focus” button on the right control panel to reset the objective to the default value.

Use the z button on the right control panel and try to make the rings disappear.

Increase the magnification to 125K. Again use the trackball to center the beam, condense the beam and use the z buttons again to make the rings go away.

Next we must center the C2 aperture.

Set the C3 aperture to position 3.

Use the trackball to center the beam. Use the intensity control to condense the beam and then spread the beam. Notice that the beam expands off center indicating the C2 aperture should be adjusted.

Rotate the C2 dials to center the beam, one dial moves it in the x and the other dial moves centers it in the y direction

Again condense the beam with the intensity button and open it to confirm that the beam remains on center.

Next we adjust the condenser stigmatism. Click on “tune”, and “condenser” from the stigmator menu.

Use the “Multifunction knobs (MF)” on either side of the scope to adjust the condenser stigmatism in both x and y directions to ensure the beam is round and expands concentrically.

After you are finished, be sure to click “none” otherwise the multifunction will continue to control the stigmatism.

Now we do direct alignments.

Select “Tune”, “user interface”, “Direct alignments” and beam tilt ppX.

Notice that there appears to be two beams. Adjust multifunction X and Y until the two beams merge to one.

Click Done to save this setting.

Repeat for beam tilt ppY, and adjust the multifunction X and Y knob until there is one beam image.

Select “Beam shift and center the beam it with the multifunction key and then click Done.

This saves the position of the beam so it can be recalled later if it gets lost. The trackball position can not be saved.

Select "Rotation center".

The beam begins to wobble and the image moves. Adjust the multifunction dial until the movement of the image is minimized and the edge appears simply to go in and out of focus.

Click done to finish the direct alignments

Click Gun, FEG registers. Type a name like "TEM" and save the alignment settings for future use.

Now we go to the digital microscope software to record an image. Click the screen lift button to lift the phosphor screen and direct the beam to the CCD camera. Replace the screen cover.

On the User interface software click "camera", CCD/TV camera, under controller select GatanCCD

Click "Insert" and it will turn yellow to show it is active.

Click "Search" and the image appears on the digital micrograph software.

Click "Process, 'Live/ FFT to help focus the sample. If the circle in the FFT is not round it means there is objective stigmatism.

Go back to control software and click Tune, Objective and use the multifunction knob to adjust the objective stigmatism until the FFT circle is round.

Adjust the " Focus Knob" until the image is in focus.

Go back to microscope control software, camera tab, ccd/tv and click "acquire.

To save the image in the digital microscope click the tool icon in the lower right. This brings up a new window.

Select "save numbered, click "browse, select "ntuf users, select your folder. In the dialog select "Build using string", and type in a base file name and an index number.

You can choose the default GATAN format, or tiff or jpg format.

The next time you click the "123" icon it will save the current image using the same file prefix with an incremented number.

To prepare another picture go to the microscope control, "camera, and click "Search.

On the Digital Microscope screen you will see a live image.

You can scroll the field of view using the joystick. The CCD builds an image by integrating repeated scans.

To change the magnification it is also necessary to adjust the intensity. Use the phosphor screen to avoid damaging the CCD camera with a too- intense beam.

The CCD has a 35mm field of view so your subject needs to fit within the inner circle on the phosphor screen.

Repeatedly adjust the magnification and intensity.

Lift the phosphor screen and replace the cover.

Higher magnification is more sensitive to objective stigmatism so it is necessary to correct it again.

In microscope control select “tune, “objective”, and then use the multifunction knob to make the FFT image round.

The ring of spots is the diffraction pattern from the gold nanoparticles.

After the correction click “None” to disable the stigmatism adjustment.

At high magnification it is best to use the multifunction dial to move the image around rather than moving the sample.

Adjust the focus step with the dial collar and use the smaller dial to fine-tune the focus. Use smaller steps at higher magnification.

In phase contrast mode the crystal lattice of the gold particle can be seen.

When you are finished go to the microscope control, Camera and click Insert to take the camera out.

Then go to Setup, click “Column Valves closed” to close the column valves and stop the beam.

Then go to Search click the arrow flap and in the reset section, click “Holder” to put the holder in the original position and angle.

When the light goes out you can pull the holder out straight until you feel a stop, rotate clockwise until you feel a stop.

Then it will be easy to pull the holder out.

Place the holder in the tube.

This TEM is able to do energy dispersive x-ray spectroscopy or EDX. It measures energy of x-rays that bounce off the surface of the sample.

For EDX use the low background double tilt holder.

In the user interface software it asks you to select specimen holder, select “FEI double tilt”.

It also asks you to connect the holder cable.

With the system pumped down and the light off, turn the holder counterclockwise and let the vacuum gently pull the holder in.

Click "In" and the EDX detector moves in.

This sample contains film layers of silicon and germanium alloys. We can do EDX in TEM mode, but in STEM, since the beam is focused probe about 1 nanometer, we can better locate the position for analysis.

In the microscope control in click STEM, , Stem imaging, and Search.

Locate an area that has the feature you want to analyze.

Under microscope control, Analysis, EDX user, click Acquire. The energy spectrum reveals peaks for silicon and germanium.

To save the EDX data click the disk icon on the right of the screen.

Locate a directory and save the emi file.